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Thermal Modification Intensity of Heat-treated Poplar Wood Part 1: Characterization and Predication of Surface Layer

Intenzitet toplinske modifikacije topolovine. Dio 1: Karakterizacija i predikcija površinskog sloja

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ABSTRACT • Wood heat treatment is an environmentally friendly method, and the heat-treated wood properties are closely related to thermal modification intensity. This study focuses on the 0-3 mm surface layer (SL) of poplar wood heat treated at 160~220 °C. The modification intensity, including surface color, hardness, chemical component and morphological changes of the SL, was evaluated. The findings of this research showed that the color difference of the poplar wood before and after heat treatment (ΔE^*_1) increased; the color difference between up-surface and down-surface of the SL (ΔE^*_2) also increased with the treatment temperature. Consequently, the surface hardness (H_R) decreased with the increase of treatment intensity. When the treatment temperature was higher than 160 °C, the up-surface and down-surface of the SL were statistically different in color. Chemical component analysis revealed that the heat treatment degrades wood components, especially the hemicellulose, and correlation analysis showed a significant correlation between the change rate of hemicellulose and the ΔE^*_1 or H_R value; the prediction functions have been established at a high confidence level of 0.99. Overall, the thermal modification intensity of the heat-treated surface layer (SL) of poplar wood varies, and the H_R and ΔE^*_1 value could be used to characterize and predict the modification intensity and degree of thermal degradation of the surface layer of heat-treated poplar wood.

KEYWORDS: heat treatment; modification intensity; hardness; color; poplar wood

SAŽETAK • Toplinska obrada drva ekološki je prihvatljiva metoda, a svojstva toplinski modificiranog drva usko su povezana s intenzitetom toplinske modifikacije. Ovo je istraživanje usredotočeno na površinski sloj (SL) od

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0 – 3 mm toplinski modificirane topolovine pri 160 – 220 °C. Istražen je utjecaj intenziteta modifikacije na boju površine, tvrdoću, kemijski sastav i morfološke promjene površinskog sloja. Rezultati su pokazali da se s povećanjem temperature modifikacije povećala razlika u boji topolovine prije i nakon modifikacije (ΔE^*_y) te razlika u boji između gornje i donje površine površinskog sloja (ΔE^*_y). Posljedično se tvrdoća površine (H_R) smanjila s povećanjem intenziteta modifikacije. Pri temperaturi modifikacije višoj od 160 °C razlika u boji između gornje i donje površine površinskog sloja statistički je značajna. Analiza kemijskog sastava otkrila je da toplinska modifikacija razgrađuje komponente drva, posebice hemicelulozu, a korelacijskom je analizom utvrđena značajna međusobna ovisnost između stupnja promjene hemiceluloze i vrijednosti ΔE^*_y i H_R . Utvrđene su funkcije predviđanja s visokom razinom pouzdanosti od 0,99. Sve u svemu, intenzitet toplinske modifikacije topolovine u površinskom sloju varira, a vrijednosti H_R i ΔE^*_y mogu se iskoristiti za karakterizaciju i predviđanje intenziteta modifikacije i stupnja toplinske razgradnje površinskog sloja toplinski modificirane topolovine.

KLJUČNE RIJEČI: toplinska obrada; intenzitet modifikacije; tvrdoća; boja; topolovina

1 INTRODUCTION

1. UVOD

Lignocellulosic materials are thought to be one of the most promising alternative resources for fossil fuels. Worldwide attention has been focused on environment-friendly material, and biomass-based functional materials have been applied in various fields (Li *et al.*, 2018; Wu *et al.*, 2021). Wood is renewable and degradable with a high strength-to-weight ratio, and it could also provide a comfortable indoor environment because of its decorative color and humidity-control properties (Zhang *et al.*, 2016).

As one of the main artificial trees in China, poplar wood could provide a large amount of timber for uses such as paper making and wood-based panels (Yang *et al.*, 2020). However, poplar wood also has many shortcomings, such as low density, dimensional stability, and durability (Korkut *et al.*, 2008), which prevents its use as solid wood material. Many modification methods like acetylation (Mantanis, 2017), resin impregnation (Xu *et al.*, 2021), densification (Srivaro *et al.*, 2021) and natural, eco-friendly chemicals (Primaningtyas *et al.*, 2021), and heat treatment (Chu *et al.*, 2020) were applied to provide a better performance and economic benefit to poplar wood and broaden its application field (Dong *et al.*, 2020). Heat treatment deepens the color of wood (Liu *et al.*, 2021) and reduces wood moisture absorption, enhances dimensional stability (Gu *et al.*, 2019), and improves biological durability (Gérardin, 2016). Therefore, demands for heat-treated wood have increased rapidly worldwide (Hill *et al.*, 2021), and its application has grown dramatically in various fields, including furniture, architectural decoration, etc. (Gurleyen *et al.*, 2017).

Studies show that heat treatment influences the chemical properties of wood through changes in the chemical content and structure of chemical components such as extractives, hemicellulose, cellulose, and lignin (Miklečić *et al.*, 2016). The mechanical properties of treated wood are affected by the reaction medium, which mainly includes steam, inert gas (nitrogen), air, hot oil,

or water (Rahimi *et al.*, 2019). High-temperature heat treatment of wood usually reduces its mechanical properties (Wu *et al.*, 2019; Widmann *et al.*, 2012), and increases overall brittleness (Hughes *et al.*, 2015; Yildiz *et al.*, 2006). Guo *et al.* (2016) used the stress-strain curve to divide the elastic and plastic regions and found that the brittleness of wood increased significantly with the increase in thermal modification intensity. Schneid *et al.* (2014) used the same method to calculate the brittleness of the heat-treated wooden chair; the result showed that the brittle was highly correlated with thermal mass loss. Fu *et al.* (2020) found that the change of chromatic value can accurately predict the mechanical properties of heat-treated wood, and that the surface color was closely related to mechanical properties (Nasir *et al.*, 2019). Lengowski *et al.* (2021) found that the hardness of the heat-treated teak wood decreased due to the degradation of amorphous polysaccharides during the heat treatment process, and that the hardness was related to thermal mass loss.

In practice, the processing performance of heat-treated wood is closely associated with the properties of the surface layer of a certain thickness, and the chemical or physical characteristics of wood surfaces affect the gluing and coating processes (Gu *et al.*, 2019; Hill *et al.*, 2021). Thermal modification intensity of the surface layer is an influential factor in determining the processing performance and quality of heat-treated wood, according to the theory of mechanical weak boundary layer (MWB) (Stehr *et al.*, 2020; Chu *et al.*, 2020). Wang (2011) studied the thermal effect of vacuum heat treatment on *eucalyptus grandis* and found that the sample dimension had a significant impact on unit mass heat absorption, leading to uneven heat transfer in the direction of thickness. Ramdane *et al.* (2015) found a significant difference in temperature with the increase of sample thickness based on transient analysis of the heat and mass transfer process. The thermal degradation intensity of wood surface layer is higher than that of the inner part of wood, because of the low thermal conductivity of wood and the heat and mass diffusion, and the moisture or contacting medium difference.

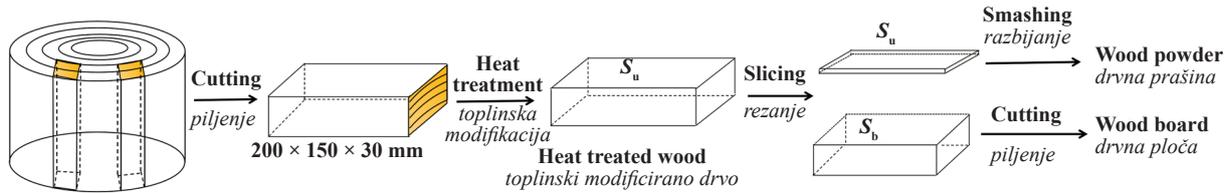


Figure 1 Flow chart of sample preparation
Slika 1. Dijagram toka pripreme uzorka

In our previous studies, it has been reported that the heat treatment process declined the surface abrasion and Shore D hardness, as well as bonding capacity (Chu *et al.*, 2020), and that an enhanced surface layer could be generated by an impregnated chemical agent and thermal compression (Chu *et al.*, 2019). However, there are few studies on the degree of thermal degradation of the heat-treated wood at 0-3 mm surface layer to the best of the authors knowledge, although there are several studies involving the overall properties of the wood (Schneid *et al.*, 2014; Costa *et al.*, 2020). This study aims to study the influence of temperature and duration on thermal modification intensity of heat-treated poplar wood at 0-3 mm surface layer and establish a potential fitting model for revealing the variation of degradation degree of the heat-treated wood regarding the surface layer. The results would provide the basis for evaluating the processability of the heat-treated poplar wood.

2 MATERIALS AND METHODS

2. MATERIJALI I METODE

2.1 Sample preparation

2.1. Priprema uzoraka

The 12-year-old Zhonglin 46 poplars (*Populus euramericana* 'zhonglin 46') were selected, and the straight part of the air-dried lumber was cut into 200 mm × 150 mm × 30 mm sapwood boards. A self-made high-temperature experiment device was used to conduct high-temperature heat treatment on the poplar wood samples, and a cistern was installed as a steam generating device.

The poplar wood samples were put evenly in the experiment box, the temperature of the inside of the box was raised from room temperature to 130 °C at a heating rate of 20 °C·min⁻¹, kept for 30 minutes, and then the box was set at a heating rate of 10 °C·min⁻¹. The internal temperature was raised to the target treatment temperature (160 °C, 190 °C, 220 °C), for 2h and 4h. The experimental group numbers were H_{160-2^h}, H_{160-4^h}, H_{190-2^h}, H_{190-4^h}, H_{220-2^h}, H_{220-4^h}, the control group was the untreated wood. Each group had ten to fifteen duplicate specimens.

The surface layer of the heat-treated poplar wood with a thickness of 3 mm was named *SL*, and its upper-

surface and bottom-surface were marked as S_u and S_b , respectively. Then, the *SL* part of the heat-treated wood was smashed into wood powder for further use.

2.2 Measurement of color and hardness of heat-treated poplar

2.2. Mjerenje boje i tvrdoće toplinski modificirane topolovine

According to the CIE1976LAB standard colorimetric system, a precision colorimeter (HP-200, Shenzhen Hampoo) was used to obtain the surface color parameters. There were 10 replicates for each experimental group, and 5 points were randomly selected on the upper-surface (S_u) and bottom-surface (S_b) of the heat-treated wood for color measurement. Color difference value ΔE^* was calculated as follows:

$$\Delta L_1^* = L_h - L \quad (1)$$

$$\Delta a_1^* = a_h - a \quad (2)$$

$$\Delta b_1^* = b_h - b \quad (3)$$

$$\Delta E_1^* = [(\Delta L_1^*)^2 + (\Delta a_1^*)^2 + (\Delta b_1^*)^2]^{1/2} \quad (4)$$

$$\Delta E_2^* = [(\Delta L_2^*)^2 + (\Delta a_2^*)^2 + (\Delta b_2^*)^2]^{1/2} \quad (5)$$

Where, L_h , a_h , b_h are the chromatic parameters of the poplar wood after HT, and L , a , b are the parameters of wood before the heat treatment; ΔL_2^* , Δa_2^* , Δb_2^* are the chromatic parameters difference of S_u and S_b on the heat-treated wood, ΔE_2^* is the color difference between S_u and S_b of the heat-treated wood on the surface layer.

Pressure ball hardness test uses a high-precision double-column universal testing machine (AG-X plus, Shimadzu, Japan), using a hemispherical steel indenter mold, in accordance with the national standard GB/T1941-2009 Wood hardness test method. The hemispherical steel indenter was pressed into the specimen surface of 2 mm at a uniform speed of 1 mm/min, and the pressing load at the pressed depth of 0.5 mm, 1.0 mm, 1.5 mm, and 2.0 mm was recorded and marked as P . Each experimental group was repeated 15 times. The surface hardness (H_R) was calculated based on the indentation load of the untreated poplar wood (Yu *et al.*, 2020).

$$H_R = P_l / P_0 \quad (6)$$

P_l – pressing load on 1 mm surface of heat-treated wood, unit: N;

P_0 – pressing load on 1 mm surface of untreated wood, unit: N.

2.3 Chemical component and morphological analysis

2.3.1. Kemijske komponente i morfološka analiza

Triplicates were used to perform quantitative chemical analysis of control and modified wood samples. According to the Chinese standards of wet chemical analysis of wood components shown in Table 1, the hemicellulose content was estimated by the difference between holocellulose and α -cellulose. Each group had three replicates, and the average value was calculated.

FT-IR analysis was performed on the samples (potassium bromide compressed tablets) by Fourier infrared spectrometer. Samples were scanned in the range of 390-4000 cm^{-1} with a 4 cm^{-1} resolution, and each sample was analyzed by 32 scans.

The experimental equipment used an XRD-6 X-ray diffractometer. The X-ray tube was a copper target and the wavelength $\lambda=1.5406$ nm. The test temperature was room temperature, the tube voltage was 40 kV, and the tube current was 30 mA. Surface sample wood powder was dried and pressed into thin sheets at room temperature. Then a 2θ diffraction intensity curve was drawn. The scanning angle 2θ ranged from 5° to 35° , the scanning rate was $2^\circ/\text{min}$, the step width was 0.01, and the diffraction intensity curve was recorded. It was output by the plotter immediately, and each sample was sampled twice, and the average value was taken.

The surface of untreated wood and heat-treated wood were selected, a SLide SLicer (SM2010R, Leica, Germany) was used to cut the surface of heat treated poplar. Scanning electron microscopy (SEM) scans of the tangential section of the wood chips were obtained using a scanning electron microscope FEG-XL30 (FEI, USA). Samples were covered by galvanic gold deposition using an MC1000 ion sputter (FEI, USA) with a current of 5 mA for 45 s. The analyses were performed with an acceleration voltage of 20 kV.

Table 1 Testing standard reference of heat-treated poplar wood

Tablica 1. Standardi za ispitivanje toplinski modificirane topolovine

Measured items <i>Mjerene sastavnice</i>	Testing standard reference <i>Ispitni standardi</i>
Moisture content <i>sadržaj vode</i>	GB/T 36,055–2018
Benzene-alcohol extractives <i>benzen-alkoholni ekstrakti</i>	GB/T 35,816–2018
Holocellulose <i>holoceluloza</i>	GB/T 35,818–2018
Cellulose / <i>celuloza</i>	GB/T 744–2004
Lignin / <i>lignin</i>	GB/T 35,818–2018

2.4 Data analysis and modeling of degradation intensity at surface layer

2.4.1. Analiza podataka i modeliranje intenziteta razgradnje površinskog sloja

The correlations between the chemical component content, ΔE_l^* , hardness, and crystallization values of untreated and heat-treated samples were calculated, and the correlation matrix was obtained using Origin software. Then, the prediction models of the wood degradation intensity at surface layer were established based on the hemicellulose change rate and the color changes before and after the heat treatment.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

3.1 Color and hardness of heat-treated wood on the surface layer

3.1.1. Boja i tvrdoća površinskog sloja toplinski modificiranog drva

Surface color and hardness are prominent properties of furniture, flooring, and other wooden materials. The heat treatment usually darkens the surface and decreases the hardness of wood, which could reflect the degradation intensity and brittleness degree of the heat-treated wood. Table 2 summarizes the chromatic values and hardness of the surface layers (SL) of heat-treated poplar.

As presented in Table 2 and Figure 2, the lightness value L^* of the SL of heat-treated poplar is significantly lower than that of untreated poplar. The red-green index a^* and yellow-blue index b^* are much higher. Among the chromatic parameters, the change in L^* value is the most obvious; it decreases with the increase of heat treatment temperature. The L^* of H_{160-2} and H_{220-2} are 84.69 and 34.58, respectively, which is 3.39 % and 60.55 % lower than that of untreated poplar. The a^* and b^* values first increased and then decreased with the increase of the heat treatment temperature; both a^* and b^* reached their maximum at the temperature of 190 °C. Heat treatment duration has a minor effect on the changing of wood surface color; the L^* value of H_{160-4} was 3.64 % lower than that of H_{160-2} . There was no notable difference in the chromatic parameters between H_{220-2} and H_{220-4} . Numerous studies have shown that the surface color of heat-treated wood becomes darker and tends to be red and yellow (Man-tanis, 2017), and that the change of surface color is closely related to the heat treatment temperature (Chen *et al.*, 2018; Cao *et al.*, 2018). The ΔE_l^* of H_{220-2} is 10.97 times higher than that of H_{160-2} .

The surface color of S_u and S_b of the SL in the same condition of heat-treated poplar is different, and the ΔE_2^* value ranges from 0.89 to 3.42, according to Table 2. The increase of ΔE_2^* value indicated that the

Table 2 Chromatic values and pressing load of surface layer of heat-treated poplar wood
Tablica 2. Kromatske vrijednosti i tlačno ispitivanje površinskog sloja toplinski modificirane topolovine

Groups Skupine		Color change Promjena boje			Pressing load at different depth, N Tlačno opterećenje pri različitoj dubini, N			
		ΔL^*	ΔE_1^*	ΔE_2^*	0.5 mm	1.0 mm	1.5 mm	2.0 mm
Control		-	-	-	77.52±5.76	166.80±4.85	254.68±11.60	368.81±13.88
H ₁₆₀₋₂	S _u	2.97	4.46	0.89	52.16±6.57	110.48±10.72	171.69±15.04	234.86±17.37
	S _b	2.32	4.34					
H ₁₉₀₋₂	S _u	24.37	27.2	2.73	32.27±5.16	83.24±5.32	158.66±9.16	240.66±17.85
	S _b	21.86**	24.52**					
H ₂₂₀₋₂	S _u	53.08	53.46	3.42	18.97±3.06	58.59±12.57	128.00±18.20	202.03±17.88
	S _b	50.24**	50.87**					
H ₁₆₀₋₄	S _u	6.05	8.58	2.33	68.53±6.52	155.62±5.67	245.93±9.68	339.83±18.54
	S _b	5.02*	6.55**					
H ₁₉₀₋₄	S _u	35.36	37.23	2.83	45.86±9.01	118.27±22.43	220.20±29.91	319.01±28.49
	S _b	33.39*	35.76					
H ₂₂₀₋₄	S _u	53.13	53.62	2.83	37.47±7.70	84.44±14.79	162.82±23.19	260.83±46.77
	S _b	50.52**	51.15**					

* and ** mean significant differences of 0.05 and 0.01 between the S_u and S_b / * i ** znači da su za S_u i S_b izmjerene značajno različite vrijednosti pri p<0,05 i p<0,01

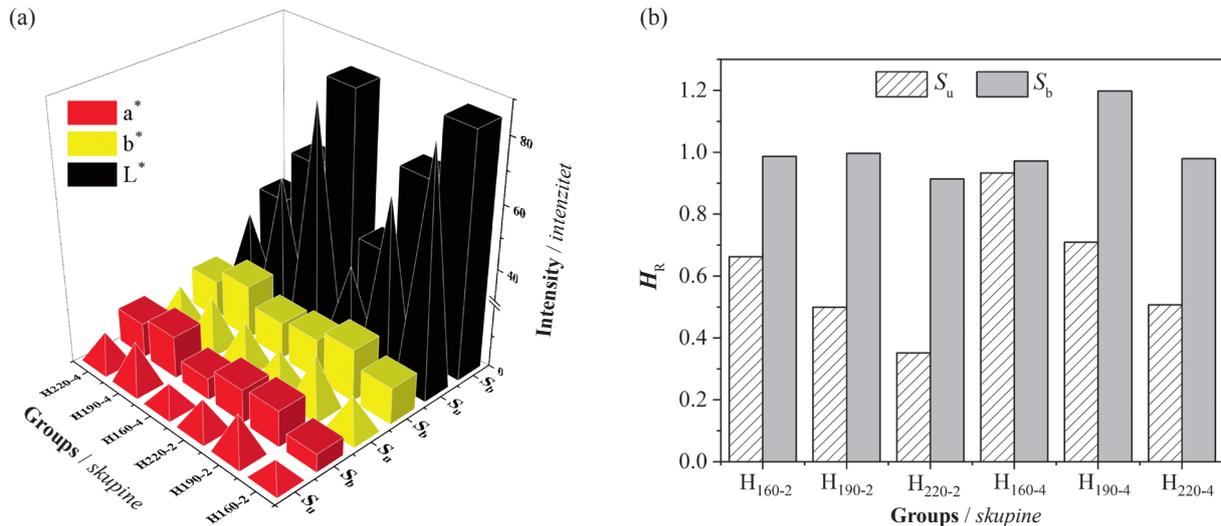


Figure 2 CIELab values (a) and hardness (b) of heat-treated poplar wood at surface layer
Slika 2. CIELab vrijednosti (a) i tvrdoća (b) površinskog sloja toplinski modificirane topolovine

degradation intensity difference between S_b and S_u increased with the increase of heat treatment temperature. Besides, the L* value of S_u is lower than that of S_b in all heat-treated groups. Variance analysis shows that in all heat-treated groups (except H₁₆₀₋₂), the ΔL^* and ΔE_1^* values are significantly different at the 0.05 level, which means the degree of thermal degradation of heat-treated poplar on the SL is different. When the heating temperature is higher than 190 °C, the uneven heating on the surface and inside leads to the difference in the degree of thermal degradation. Shi and Bao (2021) found that the extractives migrated from the core layer to the surface during the thermal modification process, which was also one of the reasons for the color difference of the wood surface layer. The possible reason for an inconsiderable difference of H₁₆₀₋₂ is

that the heat treatment is relatively low, and the SL is not significantly thermally degraded.

Figure 2 represents the H_R of heat-treated wood at the SL, which is the degree of surface plastic deformation to evaluate its ability to resist external force pressing. With the increase of the press-in depth, the ability of the compressed part to resist external force is enhanced due to the densification of the surface material. The H_R of the SL is calculated from the load when the press-in depth is 1 mm. After the heat treatment, the H_R of wood was reduced to different degrees, and the H_R value decreased with an increase in the heat treatment temperature in the duration of 2 hours. The H_R values of H₁₆₀₋₂, H₁₉₀₋₂, and H₂₂₀₋₂ decreased by 34 %, 50 %, and 65 %, respectively, compared with that of untreated wood. Previous studies stated that the hardness of wood is sensi-

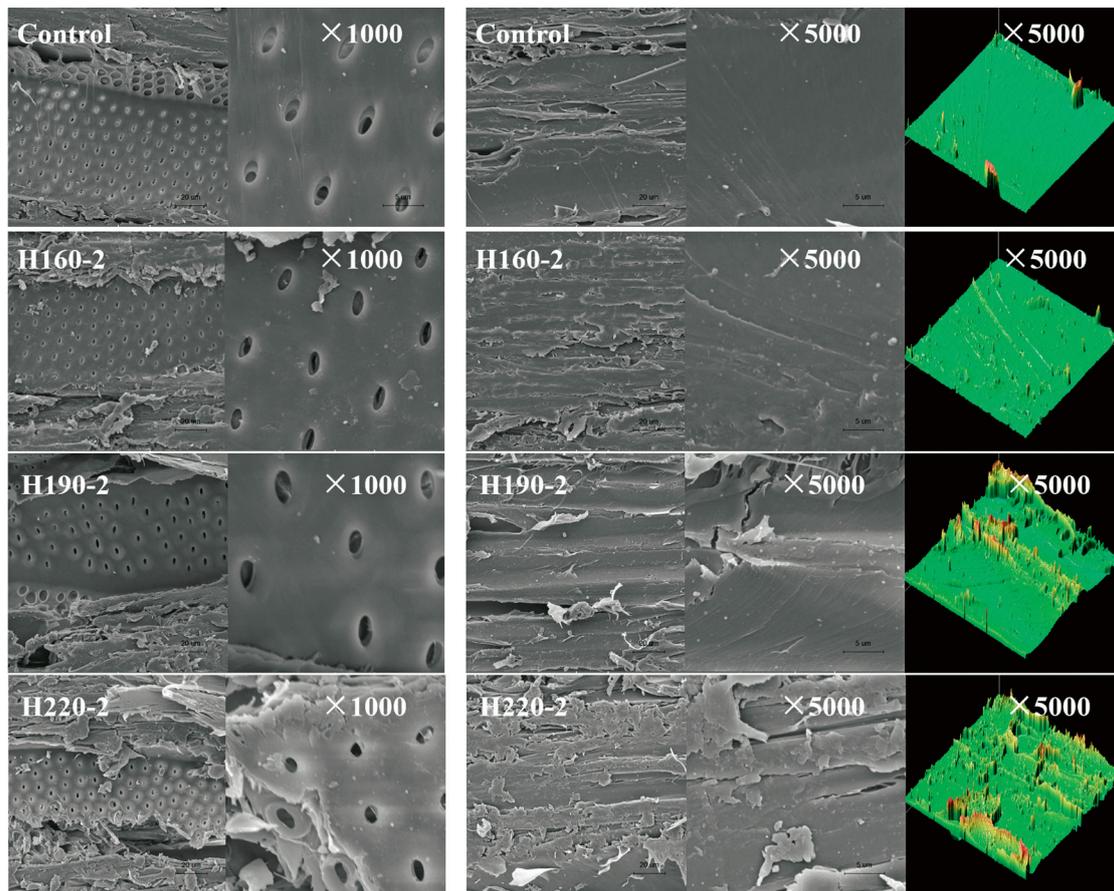


Figure 3 SEM images of heat-treated poplar surface and roughness of cell wall
Slika 3. SEM slika površine toplinski modificirane topolovine i hrapavosti stanične stijenke

tive to the change in hemicellulose content (Konnerth *et al.*, 2010; Borůvka *et al.*, 2018). Hemicellulose acts as a cell wall-filling component and its degree of degradation increases with the increase of the degree of heat treatment. The H_R value of the *SL* decreased with the increase of heat treatment temperature when the duration was 4 hours, as can be seen in Figure 2. However, the H_R value of the *SL* on H_{160-4} , H_{190-4} , and H_{220-4} was higher than that of H_{160-2} , H_{190-2} , and H_{220-2} , respectively. It could be explained by the comprehensive effects of the changing on crystallinity and the cross-linked lignin network during the long duration of the heat treatment process. When poplar wood was treated at 160 °C, the degradation of wood cell wall components was relatively low, and the crystallinity of wood increased with the HT duration, which caused the increase in H_R . When the heating temperature is higher, the crystallinity of wood decreases with the heat treatment duration, and the relative content of lignin increases with the degradation of hemicellulose and cellulose. In addition, the degradation products of hemicellulose and lignin also form cross-linked lignin networks, which leads to an increase in H_R (Altgen *et al.*, 2018). Xi (2018) found that crystallinity and lignin contents also have a great influence on wood hardness.

Existing research also showed that the destruction of the cell wall structure reduces the hardness of the

heat-treated wood (Aytekin *et al.*, 2021; Chu *et al.*, 2020). The surface cell wall and the roughness of the heat-treated wood were observed by scanning electron microscope as shown in Figure 3. It can be seen that the untreated wood surface was smooth, and the cell wall structure was undamaged. When the poplar wood was treated at 160 °C, the surface and pit structure did not change significantly. The roughness analysis showed that the sample surface was relatively smooth. So, the low-temperature heat treatment did not have a significant indigenous effect on the cell wall structure. With the increase in heat treatment temperature, the surface of H_{190-2} and H_{220-2} was cracked and gradually expanded, and some cell walls were stratified. The pit membrane deforms, and the pits become hollow and rupture, resulting in cracks and shedding. The increase of burrs on the surface made the heat-treated wood rougher, and the cell wall structure was damaged and collapsed. The cell wall structure of H_{220-2} was significantly changed and severely damaged mainly due to further degradation of hemicellulose and partial pyrolysis of lignin at the high temperature of 220 °C (Budakçı *et al.*, 2016). Wang (2017) found that the changes in the pit structure were due to a pressure gradient generated by the degradation of cell wall components, which resulted in the increase of internal pressure of wood.

3.2 Chemical component changes of surface layer of heat-treated poplar wood

3.2. Promjene kemijskih komponenata u površinskom sloju toplinski modificirane topolovine

The change of the chemical composition is the basic reason for the change of properties of the heat-treated wood. Table 3 documents the results of the wet chemical analysis of the surface layer (SL) of heat-treated poplar wood.

Among all wood components, hemicellulose is the most severely degraded by thermal treatment, and it is the main reactive substance during the heat treatment process; this result is consistent with the study of Dubey *et al.* (2012). Hemicellulose showed a downward trend with the increase of heat treatment temperature and duration; the hemicellulose contents of H_{160-4} , H_{190-4} , and H_{220-4} were 31.42 %, 21.75 %, and 15.12 %, respectively. On the contrary, the extractives content increases with the increase of heat treatment temperature and duration. Especially, the content of the extractives of H_{220-4} is 10.13%, which is 3.26 times higher than that of untreated wood. Hemicellulose degrades to produce small molecules (Candelier *et al.*, 2020), which probably increases the extractives content. The content of cellulose and lignin are relatively stable, but when the heat treatment temperature is higher than 190 °C, cellulose also degrades to a certain extent (Chien *et al.*, 2018), and lignin branched-chain breaks (Yang *et al.*, 2021); the relative

content of these two components increases because of the decrease of hemicellulose.

The FT-IR spectra of the chemical functional group changes of poplar wood before and after heat treatment are shown in Figure 4. The experiment mainly analyzes the change of characteristic peaks in the range of 1800 cm^{-1} -750 cm^{-1} . There is a subtle change of characteristic peak at 1424 cm^{-1} after the HT process, which is assigned to the shear vibration peak of cellulose. Therefore, this peak could be chosen to normalize the spectrum (Lei *et al.*, 2021). Table 4 represents the ratio of each peak.

As shown in Figure 4(a), the absorbance at 1735 cm^{-1} is assigned to the hemicellulose acetyl non-conjugated carbonyl group (Wentzel *et al.*, 2019); its peak is reached relatively smoothly with the increase of heat treatment temperature mainly due to the degradation of hemicellulose. The higher the heat treatment temperature, the more severe the degradation of the hemicellulose. The absorption peak at 1163 cm^{-1} is attributed to the C-O-C stretching vibration of cellulose and hemicellulose. The increase in the intensity of this peak is mainly due to the formation of ether bonds by the hydroxyl groups on hemicellulose and cellulose during the heat treatment. However, the peak at 1163 cm^{-1} was reduced for H_{220-2} and H_{220-4} , indicating that the ether bond broke at a high heat treatment temperature of 220 °C. The change of absorption peak near 1058 cm^{-1} , which is attributed to the C-O shrinkage vibration of cellulose and hemicellulose, indicated that aldols products are formed

Table 3 Chemical components and XRD of surface layer of heat-treated poplar wood

Tablica 3. Kemijske komponente i XRD površinskog sloja toplinski modificirane topolovine

Groups Skupine	Chemical composition change, % Promjena kemijskog sastava, %				Crystallization Kristalizacija		
	Hemicellulose	Cellulose	Lignin	Benzene-alcohol extractives	$I_{002}(2\theta)$	$I_{am}(2\theta)$	CI_{XRD}
Control	34.83	46.63	23.71	2.38	22.22	18.62	42.09
H_{160-2}	32.79	47.22	23.50	2.24	22.20	18.48	44.82
H_{190-2}	25.91	47.69	20.43	4.67	22.26	18.86	51.38
H_{220-2}	17.05	44.19	26.81	8.30	22.24	18.44	56.35
H_{160-4}	31.42	46.35	21.36	2.53	22.16	18.58	45.33
H_{190-4}	21.75	49.60	20.83	5.14	22.26	18.52	51.27
H_{220-4}	15.12	46.86	27.88	10.13	22.46	18.92	53.05

Table 4 Intensity ratio of each peak after normalization treatment at 1424 cm^{-1}

Tablica 4. Omjer intenziteta svakog vrha nakon normalizacije na 1424 cm^{-1}

Groups Skupine	I1735	I1603	I1512	I1463	I1264	I1163	I1058
Control	0.865	0.801	0.737	0.974	1.840	0.904	2.560
H_{160-2}	0.782	0.824	0.725	0.944	1.620	1.577	2.542
H_{190-2}	0.773	0.864	0.733	0.962	1.553	1.538	2.659
H_{220-2}	0.709	0.847	0.810	0.985	1.518	1.385	2.234
H_{160-4}	0.816	0.810	0.741	0.966	1.425	1.598	2.534
H_{190-4}	0.846	0.787	0.757	0.978	1.375	1.551	2.603
H_{220-4}	0.707	0.900	0.833	1.013	1.360	1.387	2.280

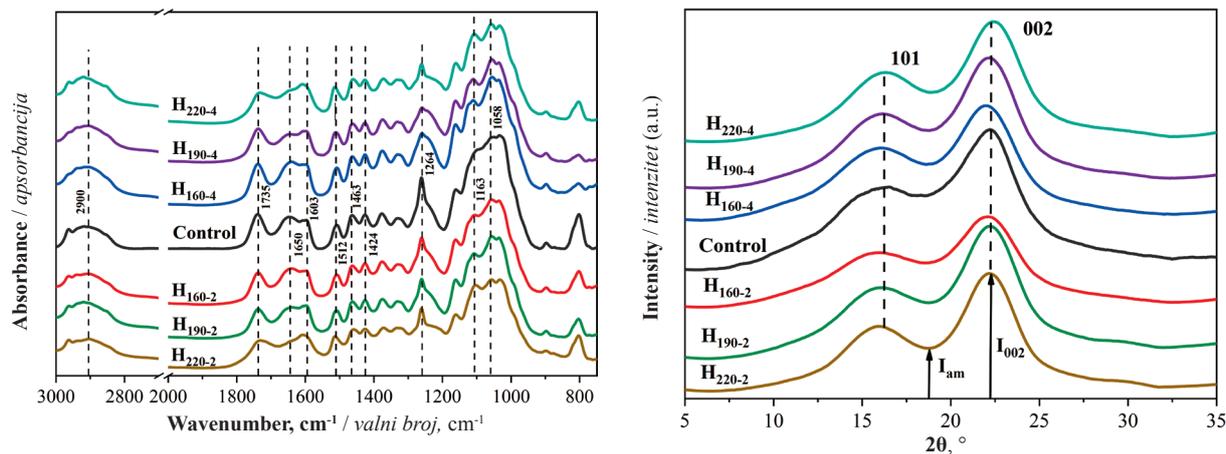


Figure 4 Infrared spectra (a) and X-ray diffraction curves (b) of surface layer of heat-treated poplar
Slika 4. Infracrveni spektar (a) i difrakcijska krivulja rendgenskih zraka (b) površinskog sloja toplinski modificirane topolovine

during the heat treatment process at relatively low temperature, while the aldols undergo condensation reaction at the high heat treatment temperature of 220 °C.

The stretching vibration peak at 1650 cm^{-1} is assigned to the C-O group in lignin, mostly located on the propyl branch of lignin. Besides, absorbance at wave numbers 1603 cm^{-1} and 1264 cm^{-1} is assigned to the conjugate stretching vibration of the C=O and the benzene ring skeleton of lignin and the stretching vibration of the G ring and the acyl-oxygen bond C=O in the lignin (Gao, 2019). With the increase of heat treatment temperature, the peak intensity at 1650 cm^{-1} and 1264 cm^{-1} decreased, while the peak intensity at 1603 cm^{-1} increased, which could also support the conclusion that the relative content of lignin increases. Furthermore, the cross-linking reaction of the lignin occurs at the same time as its thermal degradation. Absorbance at 1512 cm^{-1} and 1463 cm^{-1} is the benzene ring carbon skeleton vibration peak of lignin and the deformation vibration peak of C-H₂ in lignin and carbohydrates. The two peaks increased with the increase of the heat treatment temperature, indicating an increase in relative lignin content (Table 3). The result is consistent with that of wet chemical analysis discussed above.

The X-ray diffraction pattern is shown in Figure 4(b). Within the scanning interval of 0-35°, two prominent peaks appear on the curve. A peak valley at about $2\theta=18^\circ$ represents the scattering intensity I_{am} of the diffraction angle of the amorphous region. The peak located near $2\theta=22^\circ$ indicates the maximum intensity I_{002} of the diffraction angle of the crystal region. The crystallinity value can reflect the degree of crystal formation during the cellulose condensation process, and it is closely related to elastic modulus, impact toughness, and mechanical properties (Sun *et al.*, 2019). Table 4 lists the crystallization characteristics of SL of heat-treated poplar calculated according to I_{am} and I_{002} .

After the heat treatment, the positions of the I_{002} diffraction peaks of the samples were all around 22°,

and the crystal layer distance did not change. It means that the heat treatment did not significantly change the crystallization characteristics. When the treatment duration was 2h, the crystallinity of heat-treated wood increased from 42.09 to 56.35, with the increase in heat treatment temperature. It is worth noting that the CI_{XRD} of H_{160-4} increased slightly compared with that of H_{160-2} , and there was no evident difference between H_{190-2} and H_{190-4} . Nevertheless, a significant decline was found for H_{220-4} as the duration increased from 2h to 4h. The degradation of hemicellulose and amorphous-cellulose increases with the increase of heat treatment duration at a relatively low temperature of 160 °C, which accordingly increases the relative percentage of cellulose. Akgüil *et al.* (2007) found that the degradation products of hemicellulose could also be transformed into a crystalline area. With the increase of heat treatment temperature, part of the cellulose began to decompose, and the CI_{XRD} of H_{220-4} declined with the extension of heat treatment duration. Additionally, hemicellulose degradation generates acid substances, accelerating the deterioration of cellulose and lignin and decreases the wood crystallinity (Yang *et al.*, 2021).

3.3 Correlation analysis and prediction of degradation intensity at surface layer

3.3. Korelacijska analiza i predviđanje intenziteta razgradnje površinskog sloja

Figure 5 represents the correlation analysis of the degradation characteristics, including the color, hardness, crystallization, and the change rate of chemical components.

As shown in Figure 5(a), the SPSS analysis results show that the color difference ΔE_j^* and change rate of hemicellulose and benzene-alcohol extractives are all significantly correlated at the 0.05 statistical level. The hardness of wood was significantly correlated with the change rate of hemicellulose, while the relative crystallinity of cellulose was significantly negatively correlated with the change rate of hemicellu-

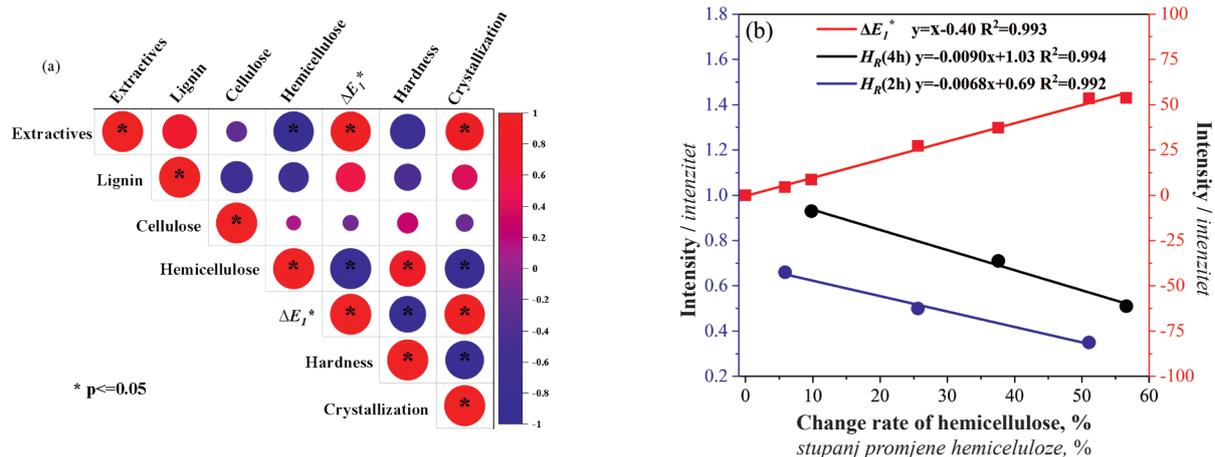


Figure 5 Correlation matrix of chemical component change rate (a) and fitting curve of change rate of hemicellulose with color difference and hardness on surface layer of heat-treated poplar wood (b)

Slika 5. Korelacijska matrica stupnja promjene kemijske komponente (a) i usklađivanje krivulja stupnja promjene hemiceluloze s razlikom u boji i s tvrdoćom površinskog sloja toplinski modificirane topolovine (b)

lose. The lower hemicellulose content, the higher crystallinity of wood; crystallinity also affects the hardness of wood. As shown in Figure 5(b), the H_R of the heat-treated poplar at the surface layer decreases with an increase in hemicellulose change rate; the functional equations of the fitted curve are $y=x-0.40$, ($R^2=0.993$); $y=-0.0068 \cdot x+0.69$, ($R^2=0.992$).

The color difference ΔE_I^* increases linearly with the changing rate of hemicellulose, and the fitted model is $y=-0.0090 \cdot x+1.03$, ($R^2=0.994$). The degree of fitting is very good, indicating that the hardness and surface color difference of the heat-treated wood surface layer is highly linearly related to the change of hemicellulose content. Therefore, they could characterize the thermal degradation of the heat-treated wood to a certain extent. This result agrees with previous studies, wherein Costa *et al.* (2020) stated that the degradation of polysaccharides could be the reason for the increased modification intensity of the high-temperature treated wood.

4 CONCLUSIONS

4. ZAKLJUČAK

The thermal modification intensity of the 160 °C, 190 °C, and 220 °C heat-treated wood at 0-3 mm surface layer was studied, and wet chemical analysis and correlation analysis were applied to reveal the relationship of surface color and hardness changes with the degree of thermal degradation of the treated wood regarding chemical changes.

The surface layer of the heat-treated wood becomes dark and consequently the surface hardness decreases with the increase of modification intensity, and the colors between the upper-surface and bottom-surface are significantly different. Heat treatment causes a change in the chemical composition of poplar wood, and

the thermal mass loss of wood mainly occurs due to hemicellulose degradation, which significantly changes the cell wall structure and could be listed as the main reason for the increase in modification intensity.

The correlation analysis shows that the color difference (ΔE_I^*) and surface hardness (H_R) have a high correlation with the change rate of hemicellulose, and a functional relationship, H_R and ΔE_I^* , could be used as a simple and rapid method to characterize the thermal modification degree of the surface layer of heat-treated wood.

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